

Physicochemical and Toxicological Profiles of Particulate Matter in Los Angeles during the October 2007 Southern California Wildfires

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To characterize the impact of the October 2007 wildfires on the air quality of Los Angeles, integrated ambient particulate matter (PM) samples were collected near the University of Southern California between October 24 and November 14, 2007. Samples were analyzed for different chemical species (i.e., water-soluble organic carbon, water-soluble elements, and several organic compounds), and the redox activity of PM was evaluated using two different assays: the dithiothreitol (DTT) and macrophage reactive oxygen species (ROS) assays. Tracers of biomass burning such as potassium and levoglucosan were elevated by 2-fold during the fire period (October 24–28), compared to the postfire period (November 1–14). Water-soluble organic carbon (WSOC) concentrations were also higher during the fire event (170 and 78 $\mu\text{g}/\text{mg}$ of PM, during fire and postfire, respectively). While the DTT activity (on a per PM mass basis) increased for samples collected during the fire event (0.024 nmol DTT/min \times μg on October 24) compared to the postfire samples (0.005 nmol DTT/min \times μg on November 14), the ROS activity appears to be unaffected by the wildfires, probably because these two assays are driven by different PM species. While the DTT assay reflected the redox potential of polar organic compounds, which are abundant in wood-smoke, the ROS assay was mainly influenced by transition metals (e.g., Fe, Cu, Cr, Zn, Ni, and V), emitted mostly by vehicular traffic and other combustion sources, but not by the wildfires.

Introduction

The semiarid Mediterranean climate of Southern California, characterized by dry and hot autumns and unusually fast (>85mph) Santa Ana winds, contributes to the occurrence of wildfires in the region, often during the fall period. Ambient levels of air pollutants including carbon monoxide (CO),

nitrogen oxides (NO_x), volatile organic compounds (VOCs), and particulate matter (PM) increase in the affected areas during these events (1, 2). Wood smoke generated from wildfires contains a large number of organic/inorganic chemicals such as aldehydes, carboxylic acids, polycyclic aromatic hydrocarbons (PAHs), resin acids, potassium, and magnesium. Many of these chemicals (e.g., PAHs and aldehydes) are toxic and potential human carcinogens (3), while some of these species (e.g., potassium and levoglucosan) are often used as tracers for the presence of wood smoke in the ambient air (4, 5). Several in vivo studies on laboratory animals found a positive correlation between wood smoke exposure and adverse health effects, such as pulmonary cell injury (6), tracheal erosion and oxidative stress (7), and allergic airway inflammation (8).

On October 20, 2007, a series of wildfires began in Southern California, ranging from Santa Barbara County to the U.S.–Mexico border, which burned more than 500,000 acres of land causing the largest mandatory evacuation in the state's history. The counties of San Diego and Los Angeles alone accounted for 91.5% of the total burnt area (9). Although the last fire was fully contained by November 9, all of the fires in the Los Angeles County were under control by October 30, 2007.

This paper presents measurements of various particulate and gaseous pollutants at a site near the University of Southern California (USC), in downtown Los Angeles, during the October 2007 fire event. Detailed chemical and toxicological characteristics of PM samples collected during the fire episode, including water-soluble organic carbon, inorganic elements, organic compounds, and reactive oxygen species, were evaluated and compared to those after the fire event, when outdoor $\text{PM}_{2.5}$ levels were mostly impacted by traffic emissions. One of our main goals was to determine the redox activity profiles of PM during these two periods and correlate them with source-specific PM constituents, in order to evaluate the potential contributions of the two major PM sources during this campaign (wildfires and vehicular traffic) to the overall PM redox properties.

Methods

Sampling Location. Sampling was conducted at the University Park Campus of USC, 2.5 km south of downtown Los Angeles. The sampling site is within 125–150 m of a major freeway (Interstate CA-110) and is representative of typical urban air quality conditions in downtown Los Angeles (10). An instrumented trailer was maintained at the site, with the aerosol sampling inlets protruding outside at 15 m from the ground. Gaseous pollutant data were taken from SCAQMD (South Coast Air Quality Management District) measurements (11) at North Main Street (Los Angeles), about 2 km northeast from the site. Satellite photos from the NASA (National Aeronautics and Space Administration) Earth Observatory taken on October 24, 2007, show the smoke plumes covering almost the entire basin, including our sampling location (Figure S1a in the Supporting Information). An analysis of the backward air mass trajectories (Figure S1c) for the Los Angeles area showed that on October 24 the air mass was traveling eastward, passing over several wildfire zones (red dots in Figure S1a) while approaching the sampling site. Satellite photos taken on October 31, 2007 (Figure S1b) indicate that the wildfires were largely contained by then, with minimum visible impacts on the air quality of the area.

Sampling Protocol. Integrated $\text{PM}_{2.5}$ samples were collected using a high volume sampler (HIQ Environmental

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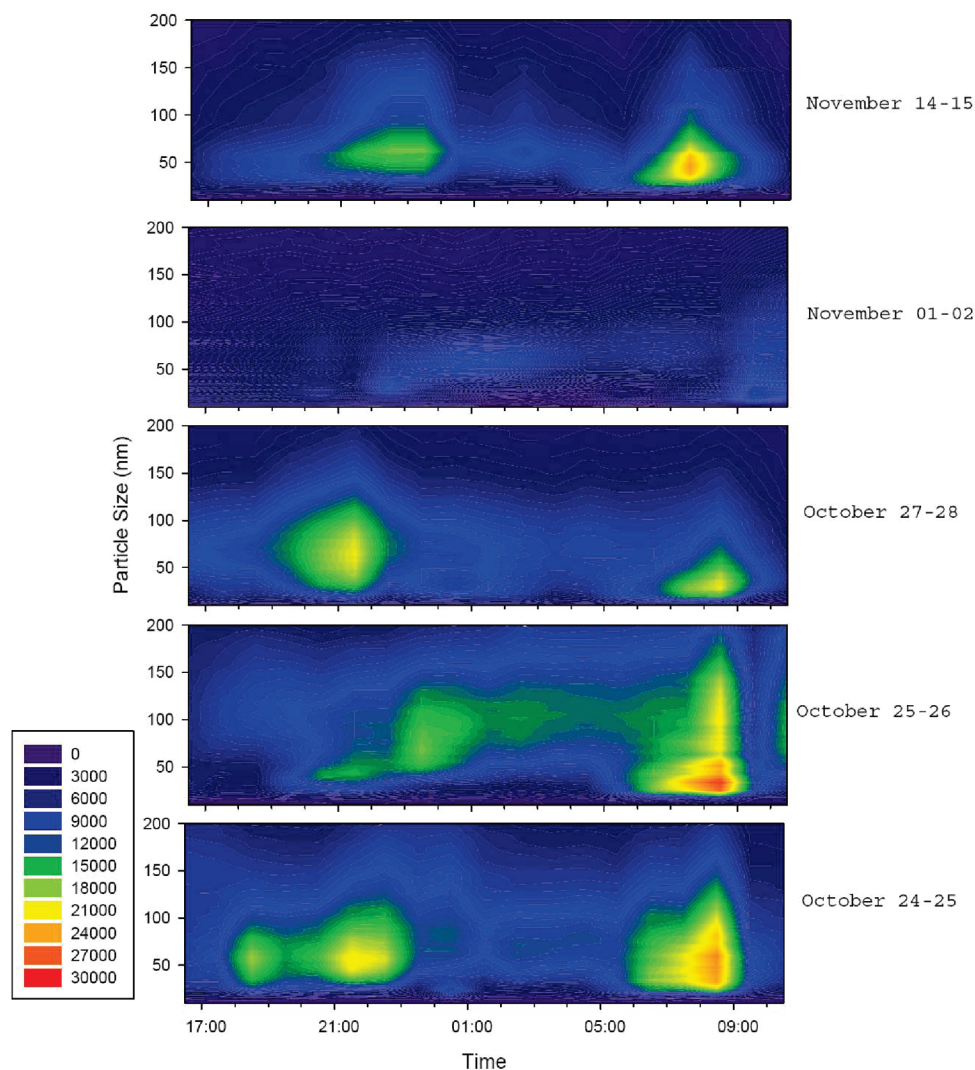


FIGURE 1. Evolution of the particle number size distribution from days characterized by wildfire emissions (October 24–25, 26–27, and 27–28) to periods not directly affected by fire (November 01–02, 14–15). Data were collected using a scanning mobility particle sizer (SMPS, Classifier model 3080 and DMA model 3081, TSI Inc., St. Paul, MN).

Products Co., CA; flow rate = 450 L/min) (12) on five different days (October 24, October 25, October 27, November 1, November 14) (Table S1). Particles were collected using Teflon-coated glass fiber filters (Pallflex, Fiberfilm T60A20 - 8 × 10 in., Pall Corp., East Hills, NY) seated in a specially designed 20 × 25 cm high-volume filter holder. Since the Los Angeles wildfires started on October 20 and continued until October 30, samples collected from October 24 to October 28 represent conditions impacted by both vehicular traffic and wildfire emissions, while the November 1 and 14 samples were considered representative of typical ambient conditions in that area, affected mostly by vehicular traffic emissions.

A scanning mobility particle sizer (SMPS model 3080 and DMA model 3081, TSI Inc., St. Paul, MN) was set up concurrently to measure the particle size distribution. The aerosol sampling flow rate was 0.3 L/min, with a scan up time of 255 s. The minimum and maximum sizes detectable at these settings are 14.1 and 736.5 nm, respectively.

Sample Analysis. All Teflon-coated glass fiber filter samples were extracted in water and then analyzed for trace elements by inductively coupled plasma-mass spectroscopy (ICP-MS) (13) and water-soluble organic carbon (WSOC) using a Shimadzu TOC-5000A liquid analyzer (14). For quantification of the organic compounds present in the particulate phase during and after the fire event, 3 during-fire samples and 2 postfire samples were composited,

respectively, and analyzed by gas chromatography/mass spectrometry (GC/MS). The redox activity of the collected particles was assessed by two different assays: (1) macrophage ROS, in vitro exposure to rat alveolar macrophage (AM) cells using dichlorofluorescein diacetate (DCFH-DA) as the fluorescent probe (15), and (2) consumption of dithiothreitol (DTT) in a cell-free system (DTT assay) (16).

Results and Discussion

Particulate Matter Characteristics. Figure 1 shows the evolution of the particle number size distribution from days during wildfires (October 24–25, 26–27, and 27–28) to the postfire period (November 01–02 and 14–15). All scans were taken from 04:30 p.m. until 10:30 a.m. of the following day to capture the effects of evening and morning traffic rush hours, which clearly caused an increase in particle number concentration between 07:00 and 11:00 p.m., and from 06:00 to 10:00 a.m. Scans collected between October 24 and 28 are characterized by a higher number of larger particles (mobility diameter ≥ 100 nm). The mode diameter was generally higher for most of the sampling time on days during fire (around 70–110 nm) than that recorded on November 01–02 and 14–15 (peak value at 60 nm). Similar results were observed during the 2003 wildfire event in Southern California (1), and previous studies also indicated that the particle number size distribution of freshly emitted wood-smoke is unimodal,

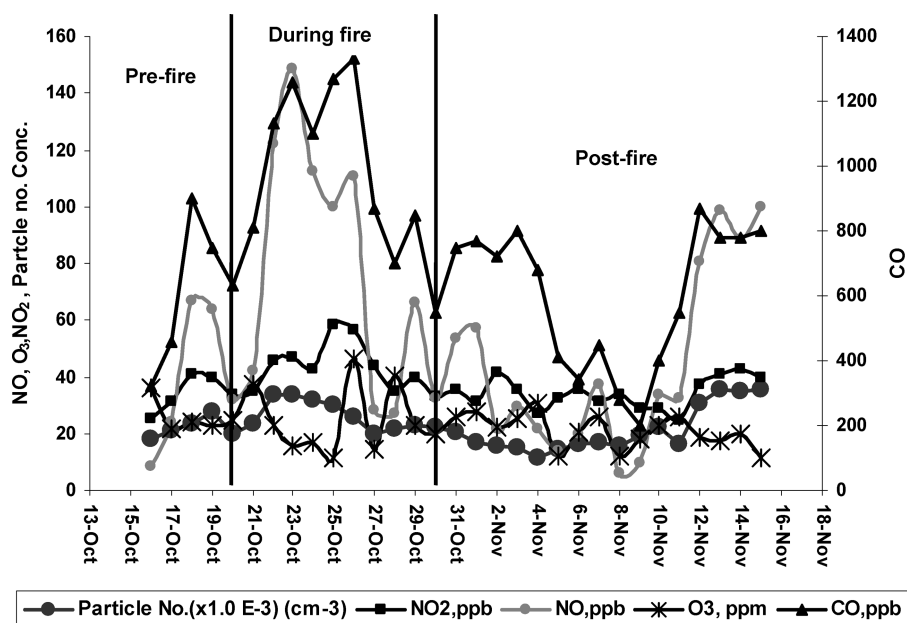


FIGURE 2. 24-h averaged concentration of NO, NO₂, CO, and O₃ at North Main Street (Los Angeles) and particle number concentration at the USC site (also 24-h average) before, during, and after the fire period.

TABLE 1. Average Fractional Concentration of Water-Soluble Elements (ICP-MS) in Ambient Particles Collected at the USC Site During (October 24, 26 and 27, 2007) and After (November 1 and 14, 2007) the Fire Event. Ratios between During- and Post-Fire Concentrations Are Also Reported. Statistically Significant Levels and Ratios (at $p \leq 0.10$) are Shown in Bold Letters

element	during fire		post fire		elevation ratio (during fire/post fire)	typical ambient levels at the sampling site ($\mu\text{g/g}$ of PM) (19)	
	average value, $\mu\text{g/g}$ of PM	standard deviation	average value, $\mu\text{g/g}$ of PM	standard deviation		average value, $\mu\text{g/g}$ of PM	standard deviation
Ti	7.11	0.94	2.13	1.11	3.34	2.89	0.65
Cu	631.73	546.24	236.10	80.42	2.68	275.23	47.44
Mn	334.75	134.77	153.49	69.72	2.18	102.52	2.00
Cd	4.00	2.58	1.85	0.71	2.16	3.89	1.45
Mg	5860.12	1328.00	2857.33	1422.36	2.05	2672.45	982.73
P	954.06	470.36	476.40	320.41	2.00	178.81	0.18
Al	577.21	464.83	298.70	414.18	1.93	256.40	24.53
K	9887.85	2102.38	5446.63	2594.56	1.82	2244.88	77.58
Zn	1513.01	1101.37	845.08	462.17	1.79	540.77	69.90
Na	28411.30	18607.50	16205.20	1421.05	1.75	14298.66	2762.341
Co	3.50	0.56	2.23	0.07	1.57	2.42	0.23
Ca	33829.90	20785.60	26472.00	27208.10	1.28	3228.07	1981.39
Fe	639.61	360.28	524.81	713.10	1.22	590.49	34.45
Cr	7.90	0.42	6.92	0.46	1.14	4.24	2.76
Ni	29.89	17.40	28.04	5.37	1.07	35.48	9.09
Pb	13.81	11.54	13.10	16.16	1.05	21.04	5.24
Ba	968.64	499.50	944.71	1199.34	1.03	404.67	152.45
S	23087.70	9865.16	28102.50	9808.47	0.82	20158.39	4923.36
V	57.08	36.38	77.50	15.20	0.74	105.54	5.86

with average mode diameter around 100 nm (17). In addition, the particle number concentration increased during the fire event by an approximate factor of 2 (Figure 2) and, although it was also notably high on November 14–15, the mode diameter remained below 60 nm, which is typical of particles emitted mainly from vehicular sources in Los Angeles (18).

Gaseous Pollutants. Figure 2 presents 24-h average concentrations of CO, nitrogen monoxide (NO), nitrogen dioxide (NO₂), and ozone (O₃) in downtown Los Angeles for pre-, during-, and postfire periods. While the concentrations of both CO and NO increased almost 3-fold during the fire event, the O₃ and NO₂ levels were marginally affected by wildfire emissions. Phuleria et al. observed a similar trend for these gases during the October 2003 Southern California

wildfire (1). Reduced photochemical activity due to the smoke blanket over the region might explain these trends in O₃ and NO₂.

Water-Soluble Trace Elements. Table 1 shows a comparison of the average fractional concentration (expressed in $\mu\text{g/g}$ of PM) of water-soluble elements in ambient particles measured at the USC site during and after the fire event. Ambient levels of these trace elements from a previous study conducted few months earlier at the same location are also provided (19). It should be noted that the average meteorological conditions during that study were very similar to those of our postfire period (temperature ranged from 59 to 63 °F, and relative humidity was between 60 and 71%). The fractional concentrations of certain elements increased by

a factor of 2 or more between October 24 and 28. The results of one-sample *t* test revealed a statistical difference (at the 95% confidence interval, or CI) between the mean fractional concentrations of Mg, P, K, and Mn observed during and after the fire event. When a CI of 90% was considered, statistical difference was also observed for Co, Ti, and other less abundant elements (e.g., Ga, Sr, Ru, and others, not shown). Potassium, often used as a signature species for biomass combustion and a tracer of forest fires (20, 2), was one of the elements whose levels were consistently elevated during the fire episode. Interestingly, the fractional concentration of K, even after the fire event, was higher (5447 $\mu\text{g/g}$) than its typical level (2245 $\mu\text{g/g}$) at the site, which indicates a likely persistence of residual wood smoke even after the fire was ceased. This may be particularly true for the November 1 sample, whose PM mass may contain a considerable fraction of residual wood smoke constituents, including resuspended smoke-related particles from the paved roads.

Several studies have reported increased concentrations of K, P, Cl, Zn, Br, Si, Ca (21), and S (2) in ambient air affected by biomass combustion or wildfires. These particle-bound elements traversed the Los Angeles basin due to the prevailing westerly wind direction. An analysis of size-segregated biomass burning samples showed that most of these elements dominate the fine PM mode (aerodynamic diameter less than 2.5 μm), which can be transported by winds over hundreds of kilometers (2). By contrast, the concentrations of other elements (e.g., V, Fe, Ni, Cu, Zn, Ba, Pb) that typically originate from vehicular emissions (10) or other combustion sources (residual fuel oil combustion) (19) and road dust (22) were similar between samples collected during and after the fire period.

Water-Soluble Organic Carbon (WSOC). The WSOC content of ambient particles was higher during the fire event ($170 \pm 80 \mu\text{g/mg}$ of PM) compared to the postfire period ($78 \pm 35 \mu\text{g/mg}$). A one-sample *t* test confirmed a statistically significant difference (95% CI) between the mean WSOC levels observed during and after the fire event. Similar increases in the WSOC concentration were observed by Saarikoski et al. in ambient particles collected in Helsinki during a major biomass-burning episode in April–May 2006 (23). Wood smoke is rich in organic compounds (56–90% by mass, depending on the wood type and combustion conditions) (4), the majority of which (up to 99%) is oxygenated in the atmosphere and becomes water-soluble (24).

These studies (including our own) confirm that biomass burning is an important source of water-soluble organic compounds in ambient aerosols. This is of particular note in the context of global climate change, as WSOC plays an important role in the formation of cloud condensation nuclei by modifying the hygroscopicity of atmospheric aerosols (25).

Organic Constituents. Figure 3 presents the fractional concentrations of the speciated organic compounds in ambient particles collected at USC (expressed as $\mu\text{g/g}$ of PM), during and after the fire period, and also from a previous study conducted at the same site few months before (19).

The concentration of levoglucosan increased by 2-fold during the fire episode compared with the postfire period (Figure 3a). However, postfire levels of levoglucosan were also significantly higher than those typically present at that site. This may be due to the fact that levoglucosan is an extremely stable compound emitted in large quantities from the pyrolysis of wood cellulose (26), and has a long residence time in the atmosphere. It is also possible that resuspended smoke-related particles contribute to the elevated levels of levoglucosan in the post fire samples.

The concentrations of most PAHs were not affected by the fire episode, with the exception of retene, which was elevated during the fire event (6.68 $\mu\text{g/g}$) but not detected

in the postfire period (Figure 3b). Retene is known to be present in wood smoke in relatively higher amounts compared to other PAHs and is commonly used as an indicator of softwood burning (27).

Figure 3c shows an increase in some of the n-alkanes concentrations measured in PM samples collected from October 24 to 28. Although this increase was not substantial (up to a factor of 1.4) compared with the corresponding postfire levels, it was quite marked (up to a factor of 3.5) with respect to the typical levels reported by Arhami et al. (19) at the same site. Fatty acids were present in significant amounts in the “during-fire” samples, and were not quantifiable in samples collected on November 1 and November 14 (Figure 3c). Arhami et al. also showed negligible concentrations of fatty acids present in the ambient air at the same site under similar meteorological conditions (19). These hydrocarbons (n-alkanes and fatty acids) are generally emitted by the volatilization of plant waxes from the leaf surface at high temperatures, or from fossil fuel combustion (28).

Redox PM Activity. Figure 4a shows the results of the DTT assay conducted on samples collected during and after the fire event. The outcomes were reported both as nmol of DTT consumption per μg of PM (nmol DTT/min $\times \mu\text{g}$), as well as per m^3 of air (nmol DTT/min $\times \text{m}^3$). Particles collected between October 24 and 28 produced higher DTT activities per unit of PM mass than those collected on November 1 and 14. DTT activity was the highest on October 24 (0.024 nmol DTT/min $\times \mu\text{g}$) and decreased gradually with time to 0.005 nmol DTT/min $\times \mu\text{g}$ on November 14. Data by Li et al. (29) previously obtained at the same site in the absence of wildfire emissions and under similar meteorological conditions (average temperature $55 \pm 4^\circ\text{F}$, relative humidity $61 \pm 12\%$) are also shown in Figure 4a for comparison. Although the DTT activity may vary depending upon a number of factors that influence the PM chemical composition (such as season and traffic intensity), the levels reached at the peak of the fire event on October 24 are remarkably higher than those observed in previous studies at the same site (16, 29, 30).

DTT expressed in units of nmol DTT/min $\times \text{m}^3$ of air is also important in the context of population exposure to ambient PM. This parameter (DTT activity per m^3 of air) peaked on October 26 (0.80 nmol DTT/min $\times \text{m}^3$) because of a very high $\text{PM}_{2.5}$ concentration (58.85 $\mu\text{g}/\text{m}^3$; not shown) coupled with an elevated per-mass DTT activity (0.014 nmol DTT/min $\times \mu\text{g}$). Although on November 1, the per-mass activity was quite low (0.009 nmol DTT/min $\times \mu\text{g}$), the high $\text{PM}_{2.5}$ concentration (63.47 $\mu\text{g}/\text{m}^3$; not shown) led to a substantial increase in DTT activity per m^3 of air (0.60 nmol DTT/min $\times \text{m}^3$). The lower temperature and higher relative humidity on November 1 (the 24-h average values were 60°F and 80%, respectively, compared to 78°F and 31% on October 24) might have resulted in limited atmospheric mixing and, thus, higher $\text{PM}_{2.5}$ concentration. The DTT activity per m^3 of air during the fire event was consistently higher (average, 0.62 ± 0.21 nmol/min $\times \text{m}^3$) compared to that reported by Li et al. (0.27 nmol/min $\times \text{m}^3$; (29)). This discussion illustrates an important point about the relative redox activity of PM emitted from various sources: comparable activity on a per-PM mass basis may not necessarily translate into an equivalent public risk if the overall exposure varies due to changes in total PM concentration.

Figure 4b shows the results of the macrophage ROS assays on particles collected during and after the fire period. In contrast to DTT, the highest ROS activity was observed for the November 1 sample, both on per-mass and on per-volume of air basis. Although the per-mass activity was relatively high on October 26, the lower values obtained on October 24 and 27 make it difficult to associate the overall results with the occurrence of wildfires. It should be noted

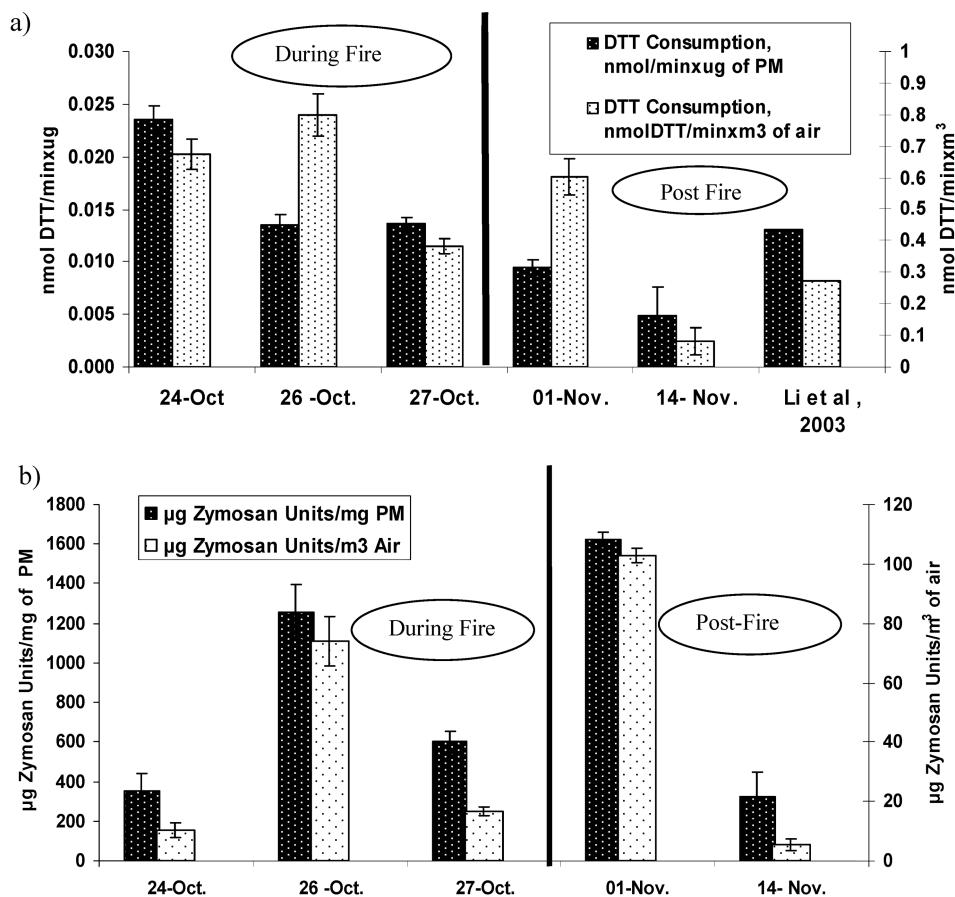


FIGURE 4. Redox activity of PM samples collected at the USC site during and after the fire period (a) expressed as nmol of DTT consumption/min, (b) expressed as the ROS response, or μg of Zymosan units.

TABLE 2. Coefficients of Statistical Determination (R^2) and Associated Levels of Significance (p -Value) for the Correlations between Selected Water-Soluble PM Constituents and Redox Activities (As Measured by the DTT and ROS Assays) for PM Samples Collected During and After the Fire Period. Statistically Significant Correlations ($R^2 \geq 0.60$; $p \leq 0.10$) are Shown in Bold

chemical constituents	correlation with DTT		correlation with ROS	
	R^2	p value	R^2	p value
WSOC	0.65	0.10	0.00	0.96
Ti	0.85	0.03	0.24	0.40
Mn	0.75	0.06	0.01	0.85
K	0.73	0.06	0.08	0.61
Mg	0.71	0.07	0.03	0.75
Co	0.71	0.08	0.56	0.14
Cr	0.61	0.12	0.84	0.03
P	0.56	0.15	0.01	0.90
Al	0.43	0.23	0.71	0.07
Ba	0.43	0.77	0.95	0.04
Pb	0.40	0.25	0.94	0.00
Fe	0.39	0.26	0.96	0.00
Cd	0.39	0.26	0.42	0.23
Ni	0.36	0.28	0.80	0.04
Zn	0.31	0.32	0.54	0.15
Na	0.29	0.36	0.33	0.31
V	0.25	0.39	0.79	0.04
S	0.22	0.42	0.93	0.08
Ca	0.15	0.49	0.21	0.47
Cu	0.08	0.65	0.04	0.75

Conversely, we observed strong associations between macrophage ROS and most of the water-soluble transition metals (Cr, V, Fe, Ni, Pb) (Table 2), consistent with earlier

studies (31, 32) showing the capability of these trace elements to induce oxidative stress in cells through the formation of ROS. The correlation between the ROS assay and elements that are not redox active (e.g., Al, Ba and S) is probably due to their common origin with transition metals (Table S3 in the Supporting Information). However, no consistent increase in ROS activity was observed for any of these species during the fire episode.

The opportunistic nature of this study constrained our sampling and data collection to a small number of measurement days, which is one of our major limitations. Although results from DTT and ROS assays may not provide definitive answers on the relative health risks associated with PM emitted from motor vehicles and wildfire (the two main PM sources during the fire episode), our study offers a novel and informative perspective on the relationship between composition and sources of atmospheric particles to their redox activities. While both assays revealed considerable redox activity of PM from traffic sources, which are ubiquitous in the Los Angeles area, the DTT assay showed additional increase in redox activity due to the contribution of the wildfires.

The DTT and ROS assays are two independent and intrinsically different types of analyses, likely driven by different particle components, and, in that respect, they should be considered complementary to each other. Both assays may thus provide important information in elucidating health risks related to PM exposure from different sources (including wood smoke emissions) and ultimately in promulgating effective control strategies to protect public health.

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Supporting Information Available

Satellite images and backward air mass trajectories for Los Angeles, sampling schedule, ambient concentration of various PM species and R^2 among those species during the wildfires. This information is available free of charge via the Internet at <http://pubs.acs.org>.

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